## QUALITY ASSURANCE / QUALITY CONTROL PROJECT PLAN

Falcon Refinery Superfund Site Ingleside San Patricio County, Texas TXD 086 278 058

Prepared for

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#### 1.0 PROJECT DESCRIPTION

This Quality Assurance/Quality Control Project Plan (QA/QCPP) has been developed by BNC Engineering, LLC (BNC) to ensure quality assurance/quality control (QA/QC) during field sample collection and analytical laboratory activities associated with removal actions at the Falcon Refinery Superfund site in San Patricio County, Texas.

Site activities may include:

- Asbestos assessment and possible abatement activities;
- Potential demolition of on-site buildings;
- Assessment and removal/disposal of oil, hazardous substances and/or pollutants and contaminants;
- Decontamination of containers, equipment, piping and buildings;
- Removal/Disposal or recycling of containers, equipment, piping and other contaminated items; and,
- Consolidation, removal/disposal of visibly contaminated soils.

All sampling and analyses will be performed pursuant to the Administrative Order on Consent for Removal Action and shall conform to EPA direction, approval and guidance regarding sampling, quality assurance/quality control, data validation and chain of custody procedures. Severn Trent Laboratories, the selected analytical laboratory for this project, is accredited under the National Environmental Laboratory Accreditation Program (NELAP) and will comply with appropriate EPA guidance.

This plan is consistent with the "Quality Assurance/Quality Control Guidance for Removal Activities: Sampling QA/QC Plan and Data Validation Procedures" (OSWER Directive No. 9360.4-01, April 1, 1990). Upon request, BNC will analyze samples submitted by the EPA for QA monitoring. Also, upon request BNC will allow EPA or its authorized representative to take split and/or duplicate samples.

EPA will be notified not less than five days in advance of any sample collection activity.

#### 1.1 SITE HISTORY

A thorough description of the past activities at the site can be found in the Removal Action Work Plan.

## 1.2 PROJECT ORGANIZATION

The BNC Project Coordinator (PC) has overall responsibility for all field activities during the implementation of the Removal Action. Severn Trent Laboratories, Inc (STL) in Corpus Christi, Texas has been selected as the primary project laboratory providing all environmental analysis.

#### 1.3 MANAGEMENT RESPONSIBILITY

<u>Stephen Halasz – Project Coordinator</u>

The PC will provide the major point of contact and control for matters concerning the project. Specifically the PC will:

- Define project objectives;
- Establish project policy and procedures to address the specific needs of the project;
- Acquire and apply technical resources as needed to ensure performance;
- Monitor and direct field personnel;
- Review work performed on each task to ensure its quality, responsiveness and timeliness;
- Approve all reports;
- Represent the project team at meetings and public hearings; and
- Approve the QA/QCPP.

#### James E. Blackwell – Project Engineer

The Project Engineer will be responsible for all mechanical aspects of the project and will be actively involved in the direction of the project. The Project Engineer will ensure that technical quality and scheduling are maintained.

#### 1.4 FIELD RESPONSIBILITY

### Theresa Nix – Field QA Officer

The Field QA Officer will be responsible for leading and coordinating day-to-day activities of the field team. The Field QA Officer will report to the Project Coordinator and specific responsibilities will include:

- Providing day-to-day coordination with the Project Coordinator;
- Developing an implementing field-related work plans;
- Coordinating and managing field staff including subcontractors;
- Performing field audits;
- Overseeing QC for technical data provided by the field staff;
- Adhering to work schedules;
- Identifying problems at the field team level and resolving difficulties in consultation with the Project Coordinator;

- Approving the QA/QCPP; and
- Participating in the final report.

#### 1.5 LABORATORY RESPONSIBILITY

Olga McDonald – STL Project Manager

STL Project Manager will have overall responsibility for QA/QC at the laboratory. In addition the Project Manager will:

- Manage and provide responses to customer inquiries related the management of the project and status of work in progress.
- Define project requirements to ensure all contract requirements are met and communicate requirements to appropriate laboratory personnel.
- Prioritize client requests based on due dates and complexity of response required.
- Manage subcontracting of samples to other STL laboratories and external laboratories after project startup phase.
- Generate and reviews final report to ensure accuracy. Facilitate corrective action when needed.
- Prepare report narratives.
- Prepare invoices to customers and follows up on accounts receivable.

Anh Tran – STL QA Officer

The Laboratory QA Officer has the overall responsibility for data after it leaves the laboratory and will communicate issues through the STL Project Manager. In addition the QA Officer will:

- Overview laboratory quality assurance;
- Overview QA/QC documentation;
- Conduct data review;
- Determine whether to implement laboratory corrective actions, if required;
- Define appropriate laboratory QA procedures; and
- Approve the QA/QCPP.

Cheyenne Whitmire – STL Sample Custodian

The Sample Custodian will report to the STL QA Officer and responsibilities will include:

- Receiving and inspecting the incoming sample containers;
- Recording the condition of the incoming sample containers;
- Signing appropriate documents'
- Verifying chain-of-custody and correctness;
- Assigning a unique identification number and customer number and entering each into the sample receiving log; and
- Control and monitor access and storage of samples.

### 2.0 QUALITY PROGRAM AND DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) specify the data type, quality, quantity, and uses needed to make decisions and are the basis for designing data collection activities. DQOs are applicable to collection activities and are based on the end use of the data being collected. DQOs will be described in detail within the individual Sampling and Analysis Plan.

## 2.1 Data Categories

The two general categories of data are defined as: (1) screening data and (2) definitive data.

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements than are necessary to produce definitive data. Sample preparation steps may be restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantitation, although the quantitation may be relatively imprecise. Physical test methods, e.g., dissolved oxygen measurements, temperature and pH measurements, moisture content, turbidity, conductance, etc., have been designated by definition as screening methods, as are immunoassay, X-Ray Fluorescence (XRF), and gas chromatography. Gas chromatography can also serve to generate definitive data.

Definitive data are generated using rigorous analytical methods (see Section 5 of this QA/QCPP), such as approved EPA reference methods. The data can be generated in a mobile or off-site laboratory. Data are analyte-specific, and both identification and quantitation are confirmed. These methods have standardized QC and documentation requirements. Definitive data are not restricted in their use unless quality problems require data qualification.

## 2.2 Data Quality Objectives

Precision, Accuracy, Representativeness, Completeness, and Comparability (PARCC) parameters are indicators of data quality. The end use of the measurement data should define the necessary PARCC

parameters. Numerical precision, accuracy, and completeness goals will be established in each SAP and will aid in selecting the measurement methods.

#### 2.2.1 Precision

Precision measures the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among: independent measurements as the result of repeated application of the same process under similar conditions. *Analytical* precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. *Total* precision is the measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations. Field duplicate samples and matrix duplicate spiked samples shall be analyzed to assess field and analytical precision, and the precision measurement is determined using the relative percent difference (RPD) between the duplicate sample results. For replicate analyses, the relative standard deviation (RSD) is determined.

## 2.2.2 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS or matrix spike sample to a control limit. For volatile and semivolatile organic compounds, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed. Analysis of performance evaluation (PE) samples shall also be used to provide additional information for assessing the accuracy of the analytical data being produced.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements.

## 2.2.3 Representativeness

Objectives for representativeness are defined for each sampling and analysis task and are a function of the investigative objectives. Representativeness shall be achieved through use of the standard field, sampling, and analytical procedures. Representativeness is also determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, and sampling locations. Decisions regarding sample locations and numbers and the statistical sampling design are documented in the Soil and Waste Sampling and Analysis Plan.

#### 2.2.4 Completeness

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples. Completeness is calculated and reported for each method, matrix and analyte combination. Completeness shall be calculated in two ways: 1) The number of valid individual analyte results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set for risk assessment; and 2) the

number of valid sample points divided by the number of planned sample points, expressed as a percentage, determines the completeness of the data set for remedial investigation/feasibility studies. For completeness requirements, valid results are all results not qualified with an "R" flag. The requirement for completeness is 95 percent for aqueous samples and 90 percent for soil samples. For any instances of samples that could not be analyzed for any reason (holding time violations in which resampling and analysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of valid results minus the number of possible results not reported. BNC will note in the QA report any significant anomalies in the data set.

The formula for the calculation of completeness for risk assessment is presented below:

% Completeness = <u>number of valid results</u> number of possible results

The formula for the calculation of completeness of a data set is presented below:

% Completeness = <u>number of valid sample points</u> number of planned sample points

### 2.2.5 Comparability

Comparability is the confidence with which one data set can be compared to another data set. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. The number of matrices that are sampled and the range of field conditions encountered are considered in determining comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions and using standard and comprehensive reporting formats. Complete field documentation using standardized data collection forms shall support the assessment of comparability. Analysis of performance evaluation (PE) samples and reports from audits shall also be used to provide additional information for assessing the comparability of analytical data produced among subcontracting laboratories. Historical comparability shall be achieved through consistent use of methods and documentation procedures throughout the project.

#### 3.0 SAMPLING LOCATIONS AND PROCEDURES

#### 3.1 Sampling and Analysis Plans

The purpose of a SAP is to provide specific guidance for field and laboratory activities associated with a task. Each SAP will include a description of the objectives of the task and corresponding DQOs. The DQO process will ensure that sampling analytical procedures meet the objectives.

For each proposed test, the SAP will include the appropriate analytes of interest, sample types and frequency, and a sample location map. The SAP will describe sample handling, control, transport, and storage procedures. Some general guidance is provided in the following subsections

#### 3.1.1 Sampling Procedures

Procedures for collecting field samples and/or data are presented in Appendix A – Field Sampling Plan. Sample collection protocols are based on EPA and industry acceptable practices. Detailed procedures will be included or referenced in each SAP, along with a description of the sampling and analysis strategy in the sampling design.

#### 3.1.2 Method of Analysis

Each SAP shall list for each analytical method the (a) method number, (b) compounds and/or elements of interest, (c) number of samples of each matrix to be collected, (d) holding times, and (e) sample locations.

## 3.1.3 Time Considerations for Transporting Samples

Samples will be taken to the analytical laboratories for analyses on the day of collection, if possible.

#### 3.1.4 Preparations of Sampling Equipment

Pre-cleaned sample containers will be secured from the analytical laboratory. Containers will remain closed until ready for use. Field sampling equipment will be cleaned before being transported to the field. Analysis of equipment rinsates will evaluate the effectiveness of field decontamination.

#### 3.1.5 Quality Control Samples

The SAP will list the number, type and matrix of field QA/QC samples. This includes trip blanks, equipment rinsates, field blanks, and field duplicates as appropriate for the medium being sampled. Definitions and requirements for QA/QC samples are outlined in Section 9.

## 3.2 Detailed Operating Procedures and Standard Operating Procedures

The SAP will include or reference, all activities that acquire environmental data or affect the quality of environmental data, including sampling, testing, drilling, groundwater monitoring well installation, decontamination, and calibration activities.

#### 3.3 Decontamination Procedures

During a sampling event, all procedures will be followed that will prevent or minimize cross contamination and thereby affect the integrity and quality of the samples. The analysis of equipment rinsates will document the effectiveness of those procedures. Specific decontamination methods will be documented in or referenced in the SAP.

#### 4.0 CONTROL DOCUMENTS

Control Documents are those that describe activities affecting data and data quality that potentially will be used as evidence. All sample identification documents and other controlled documents will be

serialized and completed with indelible ink. The following controlled sample identification documents will be used, if appropriate:

- Sample labels
- Chain-of-Custody records
- Sample analysis request sheets
- Electronic data storage devices and/or field logbooks
- Calibration logbooks
- Shipping logbooks
- SAP
- QA/QCPP
- Reports
- Laboratory data package
- Data qualification package

Sample labels and custody seals are examples of uncontrolled documents that will be used on the project

## 4.1 Sample Identification Numbers

Sample identification (ID) numbers will be assigned to each sample. The requirements of the sample ID number are that the number (a) must distinguish the sample from other similar evidence and (b) is traceable throughout the sampling and analysis process. The SAP will describe, or incorporate by reference, the sample identification scheme in detail.

## 4.2 Chain of Custody

To maintain and document sample possession, COC procedures will be followed. The purpose of the COC is to document the identity of a sample and its handling from point of collection until receipt by the laboratory. Thereafter, internal analysis is complete and laboratory QA/QC procedures confirm accuracy. The COC record will be a multiple copy form that serves as a written record of the handling of the sample. When a sample changes custody (when it is transferred from one person to another), the person receiving the sample will sign a COC record. Each change of possession will be documented. Thus, a written record tracking the handling of the sample will be established. The COC record may be combined with the sample analysis request sheet and will contain the following minimum information:

- Field sample identification number
- Signature of sample custodian
- Signature of other persons transferring custody
- Date and time of collection
- Sample type
- Signature of persons involved in the chain of possession and dates of transfer
- Inclusive dates of possession
- Suspected hazard
- Special Information (if appropriate)

The following general COC guidelines will be followed:

- A minimal number of persons will handle the samples.
- The COC record will accompany the samples to the laboratory.
- A copy of the COC record will be retained in the field file.
- Upon receipt at the analytical laboratory, all samples will be inspected for damage or tampering.
- After receipt of the sample by the laboratory, the COC record will be returned to the project file.

#### 5.0 ANALYTICAL PROCEDURES

Analytical methods shall be specified in the SAP and justified through the DQO process. Analytical methods recommended by the EPA for fixed location laboratories are listed in SW-846. The following procedures will be used to prepare and analyze soil and waste samples for this project. The Method Quantitation Limits (MQLs), QC procedures and data validation guidelines are provided. Analytical methods, method detection limits (MDL) and method quantitation limits (MQL) are presented in Table 1.

## **5.1** Preparation Methods

#### Method SW1311 - Toxicity Characteristic Leaching Procedure

Method SW1311 is used to prepare samples for determining the hazard characteristic due to leachability of organic (semivolatile and volatile) and inorganic constituents in waste or other material, which are being classified for land disposal.

QC is accomplished by preparing a toxicity characteristic leaching procedure (TCLP) blank at a rate of one blank for every 20 extractions conducted in the extraction vessel. Additional extract is prepared so a LCS may also be extracted along with MS/MSDs for each waste type (samples of similar waste types shall be batched together). One MS must be analyzed in each project analytical batch. These QA measures are in accordance with the requirements of EPA method SW1311. The MQLs for TCLP are 10X higher.

## Method SW1312 - Synthetic Precipitation Leaching Procedure

Method SW1312 is used to prepare samples for determination of the concentration of organic and inorganic constituents that are leachable from liquids, soils, waste or other various matrices.

Quality Control is accomplished by preparing a blank at a rate of one blank for every 20 extractions conducted in the extraction vessel. Additional extract is prepared so a LCS may also be extracted along with MS/MSDs for each waste type (samples of similar waste types shall be batched together). One MS must be analyzed in each analytical batch of twenty samples. The MQLs for SPLP are the same as water reporting limits for the method analytes.

## <u>Method SW3015 - Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Metals Analysis</u>

This method is used to prepare aqueous or waste samples, that contain suspended solids, for total metals determination by graphite furnace atomic absorption spectroscopy (GFAA), FLAA or ICP. The samples are digested with acid and heated in a microwave.

### Method SW3050B - Acid Digestion of Solids, Sediments, and Sludges for Metals Analysis

This method is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by ICP or, for some metals, by GFAA. A sample is digested then refluxed with acid. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

## Method SW3051 - Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils

Method SW3051 is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by FLAA or GFAA or ICP. The samples are digested with acid and heated in a microwave. A separate aliquot of the sample is dried for a total solids and/or percent moisture determination.

#### Method SM 2540G – Solids and Moisture

Percent moisture is determined for solid samples undergoing analysis for inorganic and organic analytes. The sample is weighed, dried and then re-weighed. The moisture content is used to calculate results for soil samples on a dry weight basis. All soil or sediment results and MDLs shall be reported on a dry weight basis.

#### Method SW3550A - Ultrasonic Extraction

Method SW3550A is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The sonication process ensures intimate contact of the sample matrix and the extraction solvent.

#### Method SW5030B Modified - Purge and Trap Method

Method SW5030B Modified describes sample preparation and extraction for the analysis of VOCs. The method is applicable to nearly all types of samples, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, water, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The success of this method depends on the level of interferences in the sample. Results may vary due to the large variability and complexity of matrices of solid waste samples.

An inert gas is then bubbled through the sample solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a GC column.

## <u>Method SW5035</u> - Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples

Method SW5035 is a method for analyzing VOCs in solid matrices. This method is designed for use on samples containing low levels of VOCs. This procedure may be used with any appropriate gas chromatographic analysis, including methods 8015, 8021B and 8260B. This low soil method utilizes a hermetically sealed vial, which is sealed from the time of sampling to the time of analysis. Therefore, the losses of VOCs are negligible. The success of this method depends on the level of interferences in the sample. Results may vary due to the large variability and complexity of matrices of solid waste samples.

The sample is heated to 40°C and an inert gas is bubbled through the agitated sample to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a GC column.

## Method TX1005 - Total Petroleum Hydrocarbons

This method is an n-pentane extraction followed by gas chromatography/flame ionization detection (GC/FID) analysis, which measures the concentration of hydrocarbons between nC6 and nC35. The method uses a 1:1 mixture of commercially available unleaded gasoline and diesel #2 fuel as calibration standards and the n-alkane markers, nC6, nC12, nC28 and nC35, to establish the boiling point range boundaries. However, single hydrocarbon components can be used for calibration standard. The concentration of TPH is reported as the summation of all carbon ranges, i.e., nC6 to nC35.

## <u>Method TX1006 - Characterization of nC6 to nC35 Petroleum Hydrocarbons in Environmental Samples</u>

This method uses a silica gel column fractionation of the n-pentane extract (obtained using TNRCC Method TX1005) to separate the TPH into the aliphatic hydrocarbon fraction and the aromatic hydrocarbon fraction and includes the analysis of each of these fractions by GC/FID. Quantitation is done using the TNRCC Method TX1005 calibration extended to nC35. The GC/FID analysis of the fractions separates each fraction into discrete boiling point ranges based on normal alkane markers. The concentration within each boiling point range (e.g., >nC8 - nC10 aliphatic or >nC12 - nC16 aromatic) is reported along with the total TPH concentration between nC6 and nC35.

#### 5.2 Analytical Methods

Each potential method that may be applicable to site sampling will be discussed in theis section. Presented as Table 1 are the Method Detection Limits (MDLs) and the Method Quatitation Limits (MQLs) for the laboratory. Table 2 serves to document a summary of calibration and QC procedures for each of the methods.

### EPA Method 170.1 - Temperature

Temperature measurements are made with a mercury-filled or dial type centigrade thermometer, or a thermistor. The calibration, QC, corrective action, and data flagging requirements are on figure 2. This is a field test.

## Method SW1010 – Ignitability

Method 1020A makes use of the Setaflash or Pensky Martens Closed Tester to determine the flash point of liquids that have flash points between 0° and 110°C and viscosities lower than 150 stokes at 25°C. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

## EPA Method SW1110 – Corrosivity

This test exposes steel to liquid waste to determine the corrosivity of the waste. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

## EPA Method SW9040 (Water)/SW9045 (Soil) - pH

pH measurements shall be performed for water samples using method SW9040. pH measurements of soil samples are performed using method SW9045. Measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

#### EPA Method SW9050 - Conductance

Standard conductivity meters are used. Temperature is also reported. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

#### Method SW6010B - Metals by Inductively Coupled Plasma

Samples are analyzed for trace elements or metals using method SW6020 for water and soils. Analysis for total (i.e., acid leachable) metals requires digestion of the sample. Following digestion, the trace elements are determined simultaneously or sequentially using Inductively Coupled Plasma Mass Spectroscopy (ICP-MS). The elements and MQLs for this method are listed in Table 1. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

## Method SW7000 series - Graphite Furnace Atomic Absorption for the Metals Antimony, Arsenic, Cadmium, Chromium, Lead, Selenium and Thallium

GFAA is used to measure low concentrations of metals in water and soil samples. The samples are extracted then discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is heated resistively by an electrical current. The sample solution is dried and charred to remove sample matrix components and then atomized at temperatures sufficient to vaporize the antimony. Matrix modification is used to eliminate interference effects and may also enhance the vaporization efficiency and allow lower detection limits. MQLs for this analysis are listed in Table 1. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

#### Method SW8021B - Aromatic Volatile Organics

Aromatic volatile organics in water and soil samples are analyzed using method SW8021B. This method is a purge and trap GC method using preparation method SW5030B Modified. A temperature program is used in the GC to separate the compounds. Detection is achieved by a PID and an electrolytic conductivity detector (HECD) in series. The MQLs for the analytes are presented in Table 1. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

## Method SW8260B - Volatile Organics

Volatile (or purgeable) organics in water and soil samples are analyzed using method SW8260B. This method uses a capillary column GC/mass spectrometry technique. Volatile compounds are introduced into the GC by purge and trap (SW5030B). An inert gas is bubbled through the water samples (or a soil-water slurry for soil samples) to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples with higher contaminant levels are extracted using methanol before purging. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a capillary GC column where they are separated and then detected with a mass spectrometer. The analytes detected and MQLs (using a 25 mL purge) for this method are listed in Table 1.

Calibration - The mass spectrometer is tuned daily to give an acceptable spectrum for BFB. The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 50 15 percent to 40 percent of mass 95
- mass 75 30 percent to 60 percent of mass 95
- mass 95 base peak, 100 percent relative abundance
- mass 96 5 percent to 9 percent of mass 95
- mass 173 less than 2 percent of mass 174
- mass 174 greater than 50 percent of mass 95
- mass 175 5 percent to 9 percent of mass 174
- mass 176 greater than 95 percent, but less than 101 percent of mass 174
- mass 177 5 percent to 9 percent of mass 176

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

#### Method SW8270C - Semivolatile Organics and Polynuclear Aromatic Hydrocarbons

Semivolatile organics (also known as base/neutral and acid extractables) in water and soil samples are analyzed using method SW8270C. This technique determines quantitatively the concentration of a number of SVOCs. Samples are extracted and both base/neutral and acid extracts are then concentrated through evaporation. Compounds of interest are separated and quantified using a capillary column GC/mass spectrometer. The MQLs are listed in Table 1.

The mass spectrometer is tuned every 12 hours to give an acceptable spectrum for decafluorotriphenylphosphine (DFTPP). The tuning acceptance criteria are given in the following list as an ion abundance for each specified mass:

- mass 51 30 percent to 60 percent of mass 198
- mass 68 less than 2 percent of mass 69
- mass 70 less than 2 percent of mass 69
- mass 127 40 percent to 60 percent of mass 198
- mass 197 less than 1 percent of mass 198
- mass 198 base peak, 100 percent relative abundance
- mass 199 5 percent to 9 percent of mass 198
- mass 275 10 percent to 30 percent of mass 198
- mass 365 greater than 1 percent of mass 198
- mass 441 present, but less than mass 443
- mass 442 greater than 40 percent of mass 198
- mass 443 17 percent to 23 percent of mass 442

The IS method is used for quantitation of analytes of interest. For quantitation, RFs are calculated from the base ion peak of a specific IS that is added to each calibration standard, blank, QC sample, and sample. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

#### Method SW8270C and SW8270 SIM - Polynuclear Aromatic Hydrocarbons

Methods SW8270C and SW8270 SIM are used to determine the concentration of ppb levels of selected polynuclear aromatic hydrocarbons (PAHs) in groundwater and soils by HPLC. Samples are extracted then analyzed by direct injection. Detection is by ultraviolet and fluorescent detectors. MQLs are listed in Table 1. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

#### Method TX1005 - Total Petroleum Hydrocarbons

Method TX1005 is designed to determine total concentrations of petroleum hydrocarbons (TPH) in solid and water samples using gas chromatography, with flame ionization detection (FID). The GC method is used to separate the TPH into two ranges (nC6 to nC12 and >nC12 to nC28), and a third range (>nC28 to nC35) when applicable, based on boiling points of the hydrocarbons. MQLs are listed in Table 1. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

## <u>Method TX1006 - Characterization of Nc6 to Nc35 Petroleum Hydrocarbons in Environmental Samples</u>

Method TX1006 is designed to separate and quantify the aliphatic and aromatic fractions in petroleum hydrocarbons extracted soil and water samples. The separation is based on approximate carbon number/boiling ranges with respect to n-alkane markers from n-hexane (nC6) to n-pentatriacontane (nC35). This method is to be used in conjunction with TNRCC Method 1005, which is used for the determination of total petroleum hydrocarbons. Gas chromatography (GC) is used for separation with

flame ionization (FID) as the mode of detection. MQLs are listed in Table 1. The calibration, QC, corrective action, and data flagging requirements are given in Table 2.

### EPA Method 600-R-93/116 – Asbestos

Method 600-R-93/116 identifies asbestos minerals by Polarized Light Microscopy and has a lower quantification limit of one (1) percent by weight. The samples may be further subjected to the point counting method following the procedures described in the EPA Method for the Determination of Asbestos in Bulk Building Materials, July 1999, Section 2.2.5.27.

#### **5.3** Method Detection Limits

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The laboratory shall establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory shall revalidate these MDLs at least once per twelve month period.

Laboratories participating in this work effort shall demonstrate the MDLs for each instrument, including confirmatory columns, method of analysis, analyte, and matrix (i.e., water and soil) using the requirements in 40 CFR 136, Appendix B, or by the following instructions:

- (1) Estimate the MDL using one of the following:
  - a) the concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5, or
  - b) the concentration equivalent of 3 times the standard deviation of replicate measurement of the analyte in reagent water, or
  - c) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve).
- (2) Analyze seven replicates of a matrix spike (ASTM Type II water for aqueous methods, Ottawa sand for soil methods) containing the analyte of interest at a concentration three to five times the estimated MDL.
- (3) Determine the variance (S<sup>2</sup>) for each
- (4) Determine the standard deviation (s) for each analyte as follows:

$$\mathbf{s} = (S^2)^{1/2}$$

(5) Determine the MDL for each analyte as follows:

$$MDL = 3.14(s)$$

(Note: 3.14 is the one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using 7 samples)

## 5.4 Reporting Limits and Sample Quantitation Limits

The laboratories participating in this work effort shall compare the results of the MDL demonstrations to the reporting limits (RLs) for each method. The MQLs cannot be less than 2.3 times the corresponding MDL. The laboratories shall also verify RLs by including a standard at or below the MQL'S as the lowest point on the calibration curve. For those results falling between the MDL and the MQL'S, an "J" flag shall be applied to the results indicating the variability associated with the result. Reporting limits shall be met in the laboratory method blanks.

The Sample Quantitation Limit (SQL) shall take into account all sample manipulations (e.g., initial volumes and/or weights, dilutions, concentrations, etc.). Sample non-detects which cannot be reported at the reporting limit due to manipulations on the sample shall be reported at the SQL. The SQL can be reported at or less than the corresponding MQL'S.

## 6.0 CALIBRATION PROCEDURES AND FREQUENCIES

Analytical instruments shall be calibrated in accordance with the analytical methods. All analytes reported shall be present in the initial and continuing calibrations, and these calibrations shall meet the acceptance criteria specified in Section 5 and associated tables. All results reported shall be within the calibration range. Records of standard preparation and instrument calibration shall be maintained. Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards shall be traceable to standard materials.

Instrument calibration shall be checked using all of the analytes listed in the QC acceptance criteria table referenced in Section 5 for the method. This applies equally to multi response analytes. All calibration criteria shall satisfy SW-846 requirements at a minimum. The initial calibration shall be verified prior to the analysis of any environmental samples. The initial calibration verification solution shall be prepared using materials prepared independently of the calibration standards and at a different concentration than that of any of the initial calibration standards but still within the bounds of the calibration curve. Acceptance criteria for the calibration verification are presented in Section 5 and associated tables. Analyte concentrations are determined with either calibration curves or response factors (RFs). For gas chromatography (GC) and gas chromatography/mass spectroscopy (GC/MS) methods, when using RFs to determine analyte concentrations, the average RF from the initial five-point calibration. The continuing calibration verification cannot be used as the laboratory control sample (LCS).

## 7.0 DATA REDUCTION, REVIEW, VERIFICATION, REPORTING, VALIDATION, AND RECORDKEEPING

The data reduction, review, reporting, and validation procedures described in this section will ensure; (1) complete documentation is maintained, (2) transcription and data reduction errors are minimized, (3) the data are reviewed and documented, and (4) the reported results are qualified, if necessary. Laboratory data reduction and verification procedures are required to ensure the overall objectives of analysis and reporting meet method and project specifications.

In each Laboratory analytical section, the analyst performing the tests shall review 100 percent of the definitive data. After the analyst's review has been completed, the following data shall be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria:

- Calibrations
- Blanks
- LCS recoveries
- Identification criteria
- Surrogate recoveries
- Internal standard areas (if applicable)
- MS/MSD %R & RPD
- Dilutions

The definitive data methods are identified in Section 5. The calibration, QC requirements, corrective action requirements, and flagging criteria required for definitive data are shown in the tables referenced in Section 5. The flagging criteria are applied by the Laboratory when acceptance criteria are not met and corrective action is not successful or corrective action is not performed. The Laboratory shall apply the appropriate data qualifying flags to each environmental field QC sample, e.g., field blanks, equipment blanks, trip blanks, field duplicates, matrix spike (MS) samples, and matrix spike duplicate (MSD) samples.

The one exception to these data flagging criteria rules applies to the tentatively identified compounds (TICs) that are identified only in the GC/MS methods. The numerical results of these TICs will always be qualified with one and only one flag for any reason, and that is the "T" flag.

The Laboratory QA section shall review 10 percent of the completed data packages, and the Laboratory project manager shall perform a sanity check review on all the completed data packages and shall ensure that all deliverables are present, qualifiers have been applied to the data, the chain-of-custody has been maintained and is documented, and that all non-conformance and other issues have been addressed in the comments to be included in the data report package. BNC's project manager shall review the entire definitive data report package with the field records and with the data objectives of the project when applying the final data qualifiers to define the usability of the definitive data. BNC shall review the field QC samples and field logs, and shall then appropriately flag any of the associated samples identified with the field QC sample. For example, each matrix spike sample would only be qualified by the Laboratory, while BNC would apply the final qualifying flag for a matrix effect to all

samples collected from the same site as the parent sample. The final qualifying flag shall be made manually in green ink by a dated initialed single line strikeout of the Laboratory flag and entry of the final flag. BNC will provide written justification for using any data flagged *R* by the Laboratory.

## 8.0 ELEMENTS OF QUALITY CONTROL

This section presents QC requirements relevant to analysis of environmental samples that shall be followed during all analytical activities for fixed-base, mobile, and field laboratories producing definitive data. The purpose of this QC program is to produce data of known quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis. This program provides a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

Laboratory QC samples (e.g., blanks and laboratory control samples) shall be included in the preparation batch with the field samples. An analytical batch is a number of samples (not to exceed 20 environmental samples) that are similar in composition (matrix) and that are extracted or digested at the same time and with the same lot of reagents. The term "analytical batch" also extends to cover samples that do not need separate extraction or digestion (e.g., volatile analyses by purge and trap) and is the number of samples (not to exceed 20 environmental samples) that are similar in composition (matrix) and analyzed sequentially. The identity of each analytical batch shall be unambiguously cross-referenced and reported with the associated sample analyses so that a reviewer can identify the QC samples and the associated environmental samples. All references to the analytical batch in the following sections and tables in this QA/QCPP refer to the analytical batch as defined here.

The type of QC samples and the frequency or use or these samples are discussed below.

#### 8.1 Laboratory Control Sample

The laboratory control sample (LCS) is analyte-free water (for aqueous analyses) or Ottawa sand (for soil analyses) spiked with all analytes listed in the QC acceptance criteria table in Section 6 and associated tables for the method. The LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. The LCS shall be carried through the complete sample preparation and analysis procedure.

The LCS is used to evaluate each analytical batch and to determine if the method is in control. The LCS cannot be used as the continuing calibration verification. One LCS shall be included in every analytical batch, or analyzed every 30 days, whichever is more frequent. The performance of the LCS is evaluated against the QC acceptance limits given in the tables in Section 5.

Whenever an analyte in an LCS is outside the acceptance limit, corrective action shall be performed. After the system problems have been resolved and system control has been re-established, all samples in the analytical batch shall be reanalyzed for the out-of-control analyte(s). When an analyte in an LCS exceeds the upper or lower control limit and no corrective action is performed or the corrective action was not effective, the appropriate flag shall be applied to all affected results. For organic analysis, surrogate and internal standards shall be evaluated to determine whether the data for individual samples is within acceptance limits and whether corrective action is required.

## 8.2 Matrix Spike / Matrix Spike Duplicate

A matrix spike (MS) and matrix spike duplicate (MSD) is an aliquot of sample spiked with known concentrations of all analytes listed in the QC acceptance criteria table in Section 6 for the method. The spiking occurs prior to sample preparation and analysis. The MS and MSD shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. The MS/MSD shall be designated on the chain of custody. The MS/MSD is used to document the bias of a method due to sample matrix. A minimum of one project sample shall be designated as an MS and MSD and shall be spiked and analyzed as part of every 20 project samples.

The performance of the MS and MSD is evaluated against the QC acceptance limits given in the tables. If either the MS or the MSD is outside the QC acceptance limits, the analytes in all related project samples shall be qualified according to the data flagging criteria.

## 8.3 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but that are not normally found in environmental samples.

Surrogates are used to evaluate accuracy, method performance, and extraction efficiency. Surrogates shall be added to environmental samples, controls, and blanks, in accordance with the method requirements.

Whenever a surrogate recovery for compounds with similar retention times is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been re-established, the sample shall be re-extracted / re-digested and reanalyzed. If corrective actions are not performed or are not effective, the appropriate validation flag shall be applied to the sample results.

#### 8.4 Internal Standards

Internal standards (ISs) are measured amounts of certain compounds added after preparation or extraction of a sample. They are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects. ISs shall be added to environmental samples, controls, and blanks, in accordance with the method requirements. When the IS results are outside of the acceptance limits, corrective actions shall be performed. After the system problems have been resolved and system control has been re-established, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions are not performed or are ineffective, the appropriate validation flag shall be applied to the sample results.

#### **8.5** Retention Time Windows

Retention time windows are used in GC and high performance liquid chromatography (HPLC) analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in SW-846 Method 8000A.

When the retention time is outside of the acceptance limits, corrective action shall be performed. After the system problems have been resolved and system control has been re-established, all samples analyzed since the last acceptable retention time check shall be reanalyzed. If corrective actions are not performed, the appropriate validation flag shall be applied to the sample results.

## 8.6 Interference Check Sample

The interference check sample (ICS), used in inductively coupled plasma (ICP) analyses only, contains both interfering and analyte elements of known concentrations. The ICS is used to verify background and interelement correction factors. If the instrument is capable of showing over correction, as a negative, then ISC will not be required. Also, if analyses of ICS on 5 consecutive days are within acceptance criteria, then analysis of ICS can be performed on a weekly basis. After any system problems have been resolved and system control has been re-established, the ICS shall be reanalyzed. If the ICS results are acceptable, all affected samples shall be reanalyzed. If corrective action is not performed or the corrective action was ineffective, the appropriate validation flag shall be applied to all affected results.

#### 8.7 Method Blank

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank shall be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process. A method blank shall be included in every SSF analytical batch.

The presence of analytes in a method blank at concentrations equal to or greater than the MQL'S indicates a need for corrective action. Corrective action shall be performed to eliminate the source of contamination prior to proceeding with analysis. After the source of contamination has been eliminated, all samples in the analytical batch shall be re-extracted / re-digested and reanalyzed. No analytical data shall be corrected for the presence of analytes in blanks. When an analyte is detected in the method blank and in the associated samples and corrective actions are not performed or are ineffective, the appropriate validation flag shall be applied to the sample results.

#### 8.8 Field Blank

The field blank consists of ASTM Type II reagent grade water poured into a volatile organic compound (VOC) sample vial at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis. Field blanks are prepared only when VOC samples are taken and are analyzed for all VOC analytes.

Field blanks are used to assess the potential introduction of contaminants from field sources (e.g., gasoline motors in operation, etc.) to the samples during sample collection. Field blanks will not be used on this project.

#### 8.9 Equipment Blank

An equipment blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. If equipment is dedicated, no equipment blank is required.

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures. Collection of equipment blanks shall be at a frequency of one equipment blank per equipment type per medium per day. Equipment blanks shall be collected immediately after the equipment has been decontaminated. The equipment blank should be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

When an analyte is detected in the equipment blank the appropriate validation flag shall be applied to all sample results from samples collected.

### 8.10 Trip Blank

Trip blanks are used to assess the potential introduction of contaminants from sample containers. The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes or during the transportation and storage procedures

When an analyte is detected in the trip blank the appropriate validation flag shall be applied to all sample results from samples in the cooler with the affected trip blank. One trip blank shall accompany each cooler of samples sent to the laboratory for analysis of VOCs.

## 8.11 Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection.

Duplicate sample results are used to assess precision of the sample collection process. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest

For samples collected for laboratory analysis, field duplicates will be collected at a rate of 10 percent of the total number of samples collected during each day of sampling for each sample matrix type. The number of samples will be rounded up to the next increment of 10, such that 21 samples would require three duplicates if collected within three days. At least one field duplicate will be collected per day of sampling and will be packaged and sent to the laboratory for analysis with the other samples of the same sample matrix type.

#### 8.12 Field Replicates

A field replicate sample, also called a split, is a single sample divided into two equal parts for analysis. The sample containers are assigned an identification number in the field such that they cannot be identified as replicate samples by laboratory personnel performing the analysis. Specific locations are designated for collection of field replicate samples prior to the beginning of sample collection.

Replicate sample results are used to assess precision for evaluating the homogeneity of composite samples, the laboratory precision, and/or the performance between two or more laboratories. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the composting process required to obtain uniform samples could result in loss of the compounds of interest.

Field replicates are not planned for this project.

## **8.13** Holding Time Compliance

All sample preparation and analysis shall be completed within the method-required holding times. The holding time begins at the time of sample collection. Some methods have more than one holding time requirement (e.g., methods for pesticides, semi-volatiles, etc.). The preparation holding time is calculated from the time of sample collection to the time of completion of the sample preparation process as described in the applicable method, prior to any necessary extract cleanup and/or volume reduction procedures. If no preparation (e.g., extraction) is required, the analysis holding time is calculated from the time of sample collection to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses. In methods requiring sample preparation prior to analysis, the analysis holding time is calculated from the time of preparation completion to the time of completion of all analytical runs, including dilutions, second column confirmations, and any required reanalyses.

Holding times given in hours (i.e. 24 hours, 48 hours) are calculated to the hour. Holding times given in days (i.e. 7 days, 28 days) are calculated to the end of the appropriate calendar day.

#### 8.14 Confirmation

Quantitative confirmation of results at or above the MQL'S for samples analyzed by GC or HPLC shall be required and shall be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector is used. The result of the first column/detector shall be the result reported. If holding times are exceeded and the analyses are performed, the results shall be flagged according to the procedures as described in Section 7

Quantitative confirmation of results is not performed for the following GC methods:

Method 8021B – BTEX; limited compound list

Method 8082 – PCB; pattern matching done, therefore no confirmation performed.

#### 8.15 Standard Materials

Standard materials, including second source materials, used in calibration and to prepare samples shall be traceable to National Institute Standards and Technology (NIST), EPA, American Association of

Laboratory Accreditation (A2LA) or other equivalent SSP approved source, if available. If an NIST, EPA or A2LA standard material is not available, the standard material proposed for use shall be included in an addendum to the SAP and approved before use. The standard materials shall be current, and the following expiration policy shall be followed: The expiration dates for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first. Expiration dates for laboratory prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an un-expired standard. The laboratory shall label standard and QC materials with expiration dates.

A second source standard is used to independently confirm initial calibration. A second source standard is a standard purchased from a vendor different than the vendor supplying the material used in the initial calibration standards. The second source material can be used for the continuing calibration standards or for the LCS (but shall be used for one of the two). Two different lot numbers from the same vendor do not constitute a second source.

#### 8.16 Supplies and Consumables

The laboratory shall inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents shall be monitored by analysis of LCSs. An inventory and storage system for these materials shall assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions.

#### 9.0 PERFORMANCE/SYSTEM AUDITS

Technical systems and performance audits shall be performed as independent assessments of sample collection and analysis procedures. Audit results will be used to evaluate the ability of Severn Trent to (1) produce data that fulfill the objectives established for the program, (2) comply with the QC criteria, and (3) identify any areas requiring corrective action. The systems audit is a qualitative review of the overall sampling or measurement system, while the performance audit is a quantitative assessment of a measurement system. Full data validation is also a quantitative check of the analytical process, where all documentation and calculations are evaluated and verified.

## 9.1 Project Audits

#### 9.1.1 State/Federal Project Audits

Audits and inspections are commonly conducted for the laboratories that will analyze project samples. Reports from these audits shall be reviewed by BNC to determine whether data produced by Severn Trent will fulfill the objectives of the program.

#### 9.1.2 Technical Systems Audits

A technical systems audit is an on-site, qualitative review of the sampling or analytical system to ensure that the activity is being performed in compliance with the Sampling and Analysis Plan (SAP) specifications. Sampling and field procedures and the analytical laboratories may be audited by BNC at the beginning of the field work. The laboratory systems audit results will be used to review laboratory operation and ensure the technical procedures and documentation are in place and operating to provide data that fulfill the project objectives and to ensure that outstanding corrective actions have been addressed.

Critical items for a laboratory or field systems audit include: (1) sample custody procedures, (2) calibration procedures and documentation, (3) completeness of data forms, notebooks, and other reporting requirements, (4) data review and validation procedures, (5) data storage, filing, and record keeping procedures, (6) QC procedures, tolerances, and documentation including traceability of standards, (7) operating conditions of facilities and equipment, (8) documentation of training and maintenance activities, (9) systems and operations overview, and (10) security of laboratory automated systems.

Critical items for a sampling systems audit include: (1) calibration procedures and documentation for field equipment, (2) documentation in field logbooks and sampling data sheets, (3) organization and minimization of potential contamination sources while in the field, (4) proper sample collection, storage, and transportation procedures, and (5) compliance with established COC and transfer procedures.

After each on-site audit, a debriefing session will be held for all participants to discuss the preliminary audit results. The auditor will then complete the audit evaluation and submit an audit report including observations of the deficiencies and the necessary recommendations for corrective actions. Compliance with the specifications presented in the SAP will be noted and noncompliance or deviations shall be addressed in writing by BNC with corrective actions and a time frame for implementation of the corrective actions. Follow-up audits will be performed prior to completion of the project to ensure corrective actions have been taken.

#### 9.1.3 Project-Specific Performance Evaluation Audits

Performance audits quantitatively assess the data produced by a measurement system. A performance audit involves submitting project-specific performance evaluation (PE) samples for analysis for each analytical method used in the project. BNC shall submit project-specific PE samples, if required. The project-specific PE samples are selected to reflect the expected range of concentrations for the sampling program. The performance audit answers questions about whether the measurement system is operating within control limits and whether the data produced meet the analytical QA specifications.

The project-specific PE samples are made to look as similar to field samples as possible and are submitted as part of a field sample shipment so that the laboratory is unable to distinguish between them and project samples analysis and reporting by the laboratory. This approach ensures unbiased sample analysis and reporting by the laboratory.

The critical elements for review of PE results include: (1) correct identification and quantitation of the PE sample analytes, within project specifications, (2) accurate and complete reporting of the results, and (3) measurement system operation within established control limits for precision and accuracy.

The concentrations reported for the PE samples shall be compared to the known or expected concentrations spiked in the samples. The percent recovery shall be calculated and the results assessed according to the accuracy criteria for the LCS. If the accuracy criteria are not met, the cause of the discrepancy shall be investigated and a second PE sample shall be submitted. BNC will notify the project staff of the situation at the earliest possible time.

#### 10.0 PREVENTIVE MAINTENANCE

A preventive maintenance program shall be in place to promote the timely and effective completion of a measurement effort. The preventive maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment due to unexpected component failure. In implementing this program, efforts are focused in three primary areas: (1) establishment of maintenance responsibilities, (2) establishment of maintenance schedules for major and/or critical instrumentation and apparatus, and (3) establishment of an adequate inventory of critical spare parts and equipment.

#### **10.1** Maintenance Responsibilities

Maintenance responsibilities for equipment and instruments are assumed by the respective facility managers. The managers then establish maintenance procedures and schedules for each major equipment item. This responsibility may be delegated to laboratory personnel, although the managers retain responsibility for ensuring adherence to the prescribed protocols.

#### 10.2 Maintenance Schedule

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. Other maintenance activities are conducted as needed. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/mass spectrometry instruments, AA spectrometers, and analytical balances).

## 10.3 Spare Parts

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment downtime. The inventory includes those parts (and supplies) that are subject to frequent failure, have limited useful lifetimes, or cannot be obtained in a timely manner should failure occur.

Field sampling task leaders and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, BNC will maintain an in-house source of backup equipment and instrumentation.

#### 10.4 Maintenance Records

Maintenance and repair of major field and laboratory equipment shall be recorded in field or laboratory logbooks. These records shall document the serial numbers of the equipment, the person performing the maintenance or repairs, the date of the repair, the procedures used during the repair, and proof of successful repair prior to the use of the equipment.

#### 11.0 CORRECTIVE ACTIONS

Corrective actions, if necessary, are to be completed once. If acceptance criteria were not met and a corrective action for sample analyses was not successful or corrective action was not performed, apply the appropriate flagging criteria. Requirements and procedures for documenting the need for corrective actions are described in this section.

#### 11.1 Corrective Action Report

Problems requiring corrective action in the laboratory are documented by the use of a corrective action report. The QA coordinator or any other laboratory member can initiate the corrective action report in the event QC results exceed acceptability limits, or upon identification of some other laboratory problem. Corrective actions can include reanalysis of the sample or samples affected, re-sampling and analysis, or a change in procedures, depending upon the severity of the problem.

## 11.2 Corrective Action System

A system for issuing, tracking, and documenting completion of formal Recommendations for Corrective Action (RCA) exists for addressing significant and systematic problems. Recommendations for corrective actions are issued only by a member of the QA group, or a designee in a specific QA role. Each RCA addresses a specific problem or deficiency, usually identified during QA audits of laboratory or project operations. A RCA requires a written response from the party to whom the RCA was issued. A summary of unresolved RCAs is included in the monthly QA report to management. The report lists all RCAs that have been issued, the manager responsible for the work area, and the current status of each RCA. A RCA requires verification by the QA group that the corrective action has been implemented before the RCA is considered to be resolved. In the event there is no response to an RCA within 30 days, or if the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved.

#### 12.0 QC REPORTS TO MANAGEMENT

At a minimum during the life of the project, the QA coordinator for BNC will prepare a summary report quarterly of the status of the project, of QA/QC problems, corrective actions taken, and unresolved RCAs with recommended solutions for management. The report shall also include results from all PE samples, audit findings, and periodic data quality assessments. This report shall be submitted to BNC upon request.

Table 1
Analytes and Associated MDLs and MQLs

QAPP - Falcon Refinery Superfund Site Ingleside, San Patricio County, Texas

Analytical Method	Analyte	Soil MDL (mg/kg)	Soil MQL (mg/kg)	Water MDL (mg/L)	Water MQL (mg/L)
TX1005	C6 to C12	8.39	50	0.91	5
	>C12 to C28	8.39	50	0.91	5
	>C28 to C35	8.39	50	0.91	5
	TPH C6 to C35	8.39	50	0.91	5
TX1006	nC6 Alipahtics	8.39	50	0.91	5
	>7-8 Aliphatics	8.39	50	0.91	5
	>8-10 Aliphatics	8.39	50	0.91	5
	>10-12 Aliphatics	8.39	50	0.91	5
Γ	>12-16 Aliphatics	8.39	50	0.91	5
	>16-21 Aliphatics	8.39	50	0.91	5
	>21-35 Aliphatics	8.39	50	0.91	5
	>7-8 Aromatics	8.39	50	0.91	5
	>8-10 Aromatics	8.39	50	0.91	5
	>10-12 Aromatics	8.39	50	0.91	5
	>12-16 Aromatics	8.39	50	0.91	5
	>16-21 Aromatics	8.39	50	0.91	5
	>21-35 Aromatics	8.39	50	0.91	5
SW-846 8021B	Benzene	0.000271	0.005	0.000032	0.0005
	Ethylbenzene	0.000292	0.005	0.000043	0.0005
	Toluene	0.000959	0.005	0.00009	0.0005
	Xylenes, Total	0.000659	0.015	0.000076	0.0015
	MTBE	0.00143	0.025	0.000121	0.0025
SW-846 8270C	1,3-Dimethylnaphthalene	0.0165	0.33	0.00005	0.00005
(PAH)	2-Methylnapthalene	0.0165	0.33	0.00005	0.00005
	Acenaphthene	0.0165	0.33	0.00005	0.00005
Waters are run by 8270C	Acenaphthylene	0.0165	0.33	0.00005	0.00005
SIM	Anthracene	0.0165	0.33	0.00005	0.00005
	Benzo(a)anthracene	0.0165	0.33	0.00005	0.00005
	Benzo(a)pyrene	0.0165	0.33	0.00005	0.00005
	Benzo(b)fluoranthene	0.0165	0.33	0.00005	0.00005
	Benzo(ghi)perylene	0.0165	0.33	0.00005	0.00005
	Benzo(k)fluoranthene	0.0165	0.33	0.00005	0.00005
	Chrysene	0.0165	0.33	0.00005	0.00005

**Table 1 (Continued)** 

	Table 1 (Continued)						
		Soil MDL	Soil MQL	Water MDL	Water MQL		
Analytical Method	Analyte	(mg/kg)	(mg/kg)	(mg/L)	(mg/L)		
rinary treat tyrethou	Dibenz(ah)anthracene	0.0165	0.33	0.00005	0.00005		
	Dibenzofuran	0.0165	0.33	0.00005	0.00005		
	Fluoranthene	0.0165	0.33	0.00005	0.00005		
	Fluorene	0.0165	0.33	0.00005	0.00005		
	Indeno(123cd)pyrene	0.0165	0.33	0.00005	0.00005		
	Naphthalene	0.0165	0.33	0.00005	0.00005		
	Phenanthrene	0.0165	0.33	0.00005	0.00005		
	Pyrene	0.0165	0.33	0.00005	0.00005		
SW-846 8270C	Acenaphthene	0.0165	0.33	0.0005	0.00003		
(Routine List)	Acenaphthylene	0.0165	0.33	0.0005	0.01		
(Routine List)	Acenaphthylene Aniline	0.0165	0.33	0.0003	0.01		
		<b>-</b>			+		
	Anthracene	0.0165	0.33	0.002	0.01		
	Benzo(a)anthracene	0.0165	0.33	0.00056	0.01		
	Benzo(b)fluoranthene		0.33	0.00057			
	Benzo(k)fluoranthene	0.0165	0.33	0.00093	0.01		
	Benzo(ghi)perylene	0.0165	0.33	0.0005	0.01		
	Benzo(a)pyrene	0.0165	0.33	0.00053	0.01		
	Benzyl Alcohol	0.0165	0.33	0.0005	0.01		
	Bis(2-chloroethoxy)methane	0.0165	0.33	0.0005	0.01		
	Bis(2-chloroethyl)ether	0.0165	0.33	0.00051	0.01		
	Bis(2-chloroisopropyl)ether	0.0165	0.33	0.0005	0.01		
	Bis(2-ethylhexyl)phthalate)	0.0165	0.33	0.0005	0.01		
	4-Bromophenyl ether	0.0165	0.33	0.0005	0.01		
	Butyl benzyl phthalate	0.0165	0.33	0.00066	0.01		
	4-Chloroaniline	0.0167	0.33	0.0005	0.01		
	4-Chloro-3-methylphenol	0.0165	0.33	0.0005	0.01		
	2-Chloronaphthalene	0.0165	0.33	0.0005	0.01		
	2-Chlorophenol	0.0333	0.33	0.00097	0.01		
	4-Chlorophenyl phenyl ether	0.0165	0.33	0.0005	0.01		
	Chrysene	0.0165	0.33	0.0005	0.01		
	Dibenz(ah)anthracene	0.0165	0.33	0.0005	0.01		
	Dibenzofuran	0.0165	0.33	0.002	0.01		
	1,2-Dichlorobenzene	0.0165	0.33	0.0005	0.01		
	1,3-Dichlorobenzene	0.0165	0.33	0.0005	0.01		
	1,4-Dichlorobenzene	0.0165	0.33	0.0005	0.01		
	3,3-Dichlorobenzidine	0.0667	0.33	0.004	0.01		
	2,4-Dichlorophenol	0.0165	0.33	0.00083	0.01		
	Diethyl phthalate	0.0165	0.33	0.0005	0.01		

**Table 1 (Continued)** 

	· ·			Water	
		Soil MDL	Soil MQL	MDL	Water MQL
Analytical Method	Analyte	(mg/kg)	(mg/kg)	(mg/L)	(mg/L)
<u> </u>	Dimethyl phthalate	0.0165	0.33	0.0005	0.01
	1,3-Dimethylnaphthalene	0.0165	0.33	0.0005	0.01
	2,4-Dimethylphenol	0.0339	0.33	0.0005	0.01
	Di-n-butyl phthalate	0.0165	0.33	0.00104	0.01
	Di-n-octyl phthalate	0.02	0.33	0.004	0.01
	2,4-Dinitrophenol	0.0165	1.65	0.01	0.05
	2,4-Dinitrotoluene	0.0165	0.017	0.00055	0.01
	2,6-Dinitrotoluene	0.0165	0.017	0.0005	0.01
	Fluoranthene	0.0165	0.33	0.00053	0.01
	Fluorene	0.0165	0.33	0.0005	0.01
	Hexachlorobenzene	0.0165	0.33	0.0005	0.01
	Hexachlorobutadiene	0.0165	0.33	0.0005	0.01
	Hexachlorocyclopentadiene	0.0165	0.33	0.004	0.01
	Hexachloroethane	0.0165	0.33	0.002	0.01
	Indeno(123cd)pyrene	0.0165	0.33	0.0005	0.01
	Isophorone	0.0167	0.33	0.0005	0.01
	2-Methyl-4,6-dinitrophenol	0.0667	0.33	0.004	0.01
	2-Methylnapthalene	0.0165	0.33	0.005	0.01
	2-Methylphenol	0.0165	0.33	0.00052	0.01
	3,4-Methylphenol	0.0227	0.33	0.00121	0.01
	Napthalene	0.0165	0.33	0.0005	0.01
	o-Nitroaniline	0.0165	0.33	0.00074	0.01
	m-Nitroaniline	0.0165	0.33	0.0005	0.01
	p-Nitroaniline	0.0667	0.33	0.004	0.01
	Nitrobenzene	0.0165	0.33	0.0005	0.01
	2-Nitrophenol	0.0165	0.33	0.00106	0.01
	4-Nitrophenol	0.0667	0.33	0.004	0.01
	n-Nitrosodi-n-propylamine	0.0165	0.017	0.0005	0.01
	n-Nitrosodiphenylamine	0.0333	0.33	0.00075	0.01
	Pentachlorophenol	0.0667	1.65	0.00147	0.05
	Phenanthrene	0.0165	0.33	0.0005	0.01
	Phenol	0.0165	0.33	0.00075	0.01
	Pyrene		0.33	0.0005	0.01
	Pyridine		0.33	0.004	0.01
	1,2,4-Trichlorobenzene	0.0165	0.33	0.0005	0.01
	2,4,5-Trichlorophenol	0.0165	0.33	0.00094	0.01
	2,4,6-Trichlorophenol	0.0165	0.33	0.00087	0.01
SW-846 8260B	Acrolein	0.00814	0.05	0.00186	0.05

**Table 1 (Continued)** 

Table 1 (Continued)								
	Water							
		Soil MDL	Soil MQL	MDL	Water MQL			
Analytical Method	Analyte	(mg/kg)	(mg/kg)	(mg/L)	(mg/L)			
	Acrylonitrile	0.00998	0.05	0.0005	0.05			
	Acetone	0.00297	0.01	0.00032	0.005			
	Acetonitrile	0.00519	0.05	0.00845	0.05			
	Benzene	0.00061	0.05	0.0001	0.005			
	Bromodichloromethane	0.00043	0.005	0.00009	0.005			
	Bromoform	0.00064	0.005	0.0001	0.005			
	Bromomethane	0.00061	0.005	0.00047	0.005			
	Tert-Butyl methyl Ether	0.00061	0.005	0.00013	0.005			
	Methyl Ethyl Ketone	0.00078	0.005	0.00182	0.005			
	Carbon Disulfide	0.00122	0.005	0.00012	0.005			
	Carbon Tetrachloride	0.00037	0.005	0.0001	0.005			
	Chlorobenzene	0.00044	0.005	0.00008	0.005			
	Chloroethane	0.00068	0.005	0.00018	0.005			
	Chloroform	0.00058	0.005	0.0001	0.005			
	Chloromethane	0.00094	0.005	0.00018	0.005			
	Dibromochloromethane	0.00028	0.005	0.00012	0.005			
	1,2-Dibromoethane	0.00022	0.005	0.00012	0.005			
	Dibromomethane	0.00056	0.005	0.00013	0.005			
	Dichlorodifluoromethane	0.00084	0.005	0.00013	0.005			
	1,1-Dichloroethane	0.00079	0.005	0.00012	0.005			
	1,2-Dichloroethane	0.00067	0.005	0.00007	0.005			
	1,1-Dichloroethene	0.00094	0.005	0.00009	0.005			
	cis-1,2-Dichloroethylene	0.00084	0.005	0.00011	0.005			
	trans-1,2-Dichloroethylene	0.00093	0.005	0.00008	0.005			
	1,2-Dichloropropane	0.00108	0.005	0.00009	0.005			
	cis-1,3-Dichloropropene	0.0006	0.005	0.00009	0.005			
	trans-1,3-Dichloropropene	0.00047	0.005	0.00009	0.005			
	1,3-Dichloropropane	0.00057	0.005	0.00012	0.005			
	2,2-Dichloropropane	0.00065	0.005	0.00009	0.005			
	1,1-Dichloropropene	0.00083	0.005	0.00016	0.005			
	1,4-Dioxane	0.04	0.1	0.02841	0.1			
	Ethyl Acetate	0.00202	0.005	0.00022	0.005			
	Ethylbenzene	0.00032	0.005	0.00007	0.005			
	Ethyl Ether	0.001	0.005	0.00011	0.005			
	Ethyl Methacrylate	0.00035	0.005	0.00013	0.005			
	2-Hexanone	0.00108	0.005	0.00021	0.005			
	Iodomethane	0.00138	0.005	0.00026	0.005			
	Methylene Chloride	0.00751	0.02	0.0003	0.005			

**Table 1 (Continued)** 

		C-21 MDI	C-21 MOI	Water	W-4 MOI
Analytical Method	Analyte	Soil MDL (mg/kg)	Soil MQL (mg/kg)	MDL (mg/L)	Water MQL (mg/L)
•	4-Methyl-2-pentanone	0.00053	0.005	0.00013	0.005
	Methyl methacrylate	0.00031	0.005	0.00012	0.005
	2-Nitropropane	0.00139	0.005	0.00161	0.005
	Styrene	0.00029	0.005	0.00006	0.005
	1,1,2,2-Tetrachloroethane	0.00074	0.005	0.00014	0.005
	Tetrachlorethylene	0.00049	0.005	0.00008	0.005
	Toluene	0.00048	0.005	0.00005	0.005
	1,2,3-Trichlorobenzene	0.00047	0.005	0.00015	0.005
	1,1,1-Trichloroethane	0.00041	0.005	0.00009	0.005
	1,1,2-Trichloroethane	0.0007	0.005	0.00009	0.005
	Trichloroethylene	0.00027	0.005	0.00009	0.005
	Trichlorofluoromethane	0.00063	0.005	0.00008	0.005
	1,1,2-Trichloro-1,2,2-trifluoroethane	0.00046	0.005	0.00019	0.005
	1,2,3-Trichloropropane	0.00077	0.005	0.00031	0.005
	1,2,4-Trimethylbenzene	0.00028	0.005	0.00005	0.005
	1,3,5-Trimethylbenzene	0.00044	0.005	0.00004	0.005
	Vinyl Acetate		0.005	0.00013	0.005
	Vinyl Chloride		0.005	0.00012	0.005
Xylenes, Total		0.00011	0.015	0.00014	15
SW-846	Arsenic	0.37	1	0.0037	0.01
6010B/7470/7471	Barium	0.06	1	0.0006	0.01
	Cadmium	0.011	1	0.00011	0.01
	Chromium	0.11	1	0.0011	0.01
	Lead	0.31	1	0.0031	0.01
	Mercury	0.03	0.5	0.00012	0.002
	Selenium	0.45	1	0.0045	0.01
	Silver	0.086	0.5	0.00086	0.005
SW-846 7.3/9010	Reactive Cyanide	N/A	5	N/A	N/A
SW-846 7.3/9034	Reactive Sulfide	N/A	50	N/A	N/A
SW-846 9040B/9045C	Corrosivity	N/A	0.1 pH Units	N/A	0.1 pH units
SW-846 1010	Ignitability	N/A	N/A	N/A	1 deg F

Table 2
Summary of Calibration and QC Procedures

	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Action <sup>a</sup>	Criteria <sup>b</sup>
SW-846 (3550)	Moisture	Duplicate Sample	One per 20 samples	% solid – RPD 15% RPD 30%	Correct problem, repeat measurement. If still out, flag data	Apply S to all results
E170.1	Temperature	Field Duplicate	10% of field samples	± 1 degree C	Correct problem, repeat measurement. If still out, flag data	Apply S to all results
SW9030 B	Reactive Sulfides	Field Duplicate	10% of field samples	RPD < 25%	Correct problem, repeat measurement. If still out, flag data	Apply S to all results
SW9010 B	Reactive Cyanides	Field Duplicate	10% of field samples	RPD < 25%	Correct problem, repeat measurement. If still out, flag data	Apply S to all results
SW1020 A	Ignitability	Field Duplicate	10% of field samples	RPD < 25%	Correct problem, repeat measurement. If still out, flag data	Apply S to all results
SW1110	Corrosivity	Duplicate	10% of field samples	RPD < 20%0	Correct problem, repeat measurement. If still out, flag data	Apply S to all results
	PH (water)	2- point calibration with buffers	Once per day	± 0.05 pH units for every day	If calibration is not achieved, check meter, buffer solutions and probe; replace if necessary; repeat calibration	None
SW9040		PH 7 buffer	At each sample location	± 0.1 pH units	Correct problem, recalibrate	None
		Field duplicate	10% of field samples	± 0.1 pH units	Correct problem, repeat measurement	None
	PH (soil)	2- point calibration with buffers	1 per 20 samples	± 0.05 pH	Check with new buffers; if still out, repair meter; repeat calibration check	None
		PH 7 buffer	At each sample location	± 0.1 pH units	Recalibrate	None

# Table 2 (Continued) Summary of Calibration and QC Procedures

Method	Applicable	QC Check	Minimum	Acceptance	Corrective	Flagging
1120220	Parameter		Frequency	Criteria	Actiona	Criteria <sup>b</sup>
SW9040		Field duplicate	10% of field samples	± 0.1 pH units	Correct problem, repeat measurement. If still out, repeat calibration and reanalyze samples	None
SW9050	Conductance	Calibration with KCL standard	Once per analyst	± 5%	If calibration is not achieved, check meter, standards and probe; recalibrate	Apply S to all results
	ICP/MS Metals	MS tuning sample	Prior to initial calibration and calibration verification	RSD ≤5% for all analytes	Retune instrument then reanalyze tuning solution	Apply R to all results for all analytes for all samples associated with the MS tuning
		Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
SW6020		Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected ≥ RL	Correct problem then analyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank
		Continuing calibration verification (Instrument Check Standard)	Before beginning a sample run, after every 10 samples and at the end of the analysis sequence	All analyte(s) within ±10% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 6.2.2-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
	ICP/MS Metals	Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
SW6020		Interference check solution (ICS)	At the beginning and end of an analytical run or twice during an 12 hour period, whichever is more frequent	Within ±20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS; reanalyze all affected samples	Apply R to all results for specific analyte(s) in all samples associated with the ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 6.2.3-2	Correct problem reprep and analyze the LCS and all samples in the affected USACE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive
	ICP/MS Metals	Dilution test	Each new sample matrix	1:4 dilution must agree within ±10% of the original determination	Perform post digestion spike addition	results, apply R to all non-detects  Apply J to all sample results if either of following exist: (1) new matrix check not run (2) RPD ≥10%

3.5.13	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Actiona	Criteria <sup>b</sup>
SW6020		Post digestion spike addition	When dilution test fails	Recovery within 75-125% of expected results	Dilute the sample; reanalyze post digestion spike addition	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 6.2.2-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Internal Standards (ISs)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in method SW6020, section 8.3	Apply R to all results for specific analyte(s) in all samples associated with the IS.
		MDL study	Every three months	Detection limits established shall be < the RLs in Table 6.2.2-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	None	none	none	Apply F to all results between MDL and RL
SW7421	Lead	Initial multipoint calibration (minimum 3 standards and a blank)	Daily initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration

Method	Applicable	QC Check	Minimum	Acceptance	Corrective	Flagging
Method	Parameter	,	Frequency	Criteria	Actiona	Criteria <sup>b</sup>
		Second- source calibration check standard	Once per initial daily multipoint calibration	Analyte within ±10% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte for all samples associated with the calibration
		Calibration blank	Once per initial daily multipoint calibration	No analyte detected ≥ RL	Correct problem then reanalyze calibration blank and all samples associated with blank	Apply B to all results for the specific analyte in all samples associated with the blank
SW7421		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	The analyte within ±20% of expected value	Correct problem then repeat calibration and reanalyze all samples since last successful calibration	Apply R to all results for the specific analyte in all samples since the last acceptable calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 6.2.3-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte in all samples in the associated analytical batch

	Applicable	Juni	Minimum		Corrective	Flogging
Method	Applicable Parameter	QC Check	Frequency	Acceptance Criteria	Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
	Lead	LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 6.2.3-2	Correct problem then reprep and analyze the LCS and all samples in the affected USACE analytical batch	For specific analyte in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		New matrix check; five- fold dilution test	Each new sample matrix	Five times dilution sample result must be ±10% of the undiluted sample result	Perform recovery test	Apply J to all sample results if either of following exist: (1) new matrix check not run (2) RPD ≥10%
SW7421	Lead	Recovery test	When new matrix check fails	Recovery within 85-115% of expected results	Run all samples by the method of standard addition	Apply J to all sample results (for same matrix) in which method of standard addition was not run when recovery outside of 85-115% range
		MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 6.2.3-2	none	For the specific analyte in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL

	Annliachla	Sam	Minimum			Flooring
Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW7421		MDL study	Once per 12 month period	Detection limits established shall be < the RLs in Table 6.2.3-1	none	Apply R to all results for the specific analyte in all samples analyzed
SW /421		Results reported between MDL and RL	None	None	none	Apply F to all results between MDL and RL
SW8021 B	Halogenated volatile organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	%RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second- source calibration verification	Once per five- point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Initial calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification

N. (1 )	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	<b>Action</b> <sup>a</sup>	Criteria <sup>b</sup>
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 6.2.4-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
	Halogenated volatile organics	Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
SW8021 B		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 6.2.4-2	Reprep and analyze the LCS and all samples in the affected USACE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 6.2.4 -2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
SW8021	Halogenated volatile organics	MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 6.2.4-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
В		Second- column confirmation	100% for all positive results	Same as for initial or primary column analysis	Same as for initial or primary column analysis	Apply R to the result for the specific analyte(s) in the sample
		MDL study	Once per 12 month period	Detection limits established shall be < the RLs in Table 6.2.4-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	None	none	none	Apply F to all results between MDL and RL
	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq$ 0.30° and %RSD for RFs for CCCs $\leq$ 30% and one option below	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration
SW8260 B				option 1 linear- mean RSD for all analytes ≤15% with no individual analyte RSD >30%		Apply R to all results for specific analyte(s) for all samples associated with the calibration
				option 2 linear – least squares regression r > 0.995		

	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Action <sup>a</sup>	Criteria <sup>b</sup>
				option 3 non- linear – COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)		
		Second- source calibration verification	Once per five- point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
SW8260 B		Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF $\geq$ 0.30°; and CCCs $\leq$ 20% difference (when using RFs)or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration	Apply R to all results for all samples associated with the calibration verification
				All calibration analytes within ±20% of expected value		Apply R to all results for specific analyte(s) for all samples associated with the calibration verification

	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Action <sup>a</sup>	Criteria <sup>b</sup>
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 6.2.5-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
SW8260 B	Volatile Organics	ISs	Immediately after or during data acquisition for each sample	Retention time ±30 seconds from retention time of the midpoint std. in the ICAL.  EICP area within -50% to +100% of ICAL mid-point std.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for analytes associated with the IS
В		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 6.2.5-2	Correct problem then reprep and analyze the LCS and all samples in the affected AFCEE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results
						if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

Method	Applicable Parameter	QC Check	Minimum	Acceptance	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
	rarameter	MS/MSD	One MS/MSD per every 20 project samples per matrix	Criteria QC acceptance criteria, Table 6.2.5-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or(2)%R for MS or MSD < LCL or (3) MS/MSD RPD > CL
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 6.0)	Retune instrument and verify	Apply R to all results for all samples associated with the tune
SW8260 B		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 6.2.5-2	Correct problem then reextract and analyze sample	For the samples;  if the %R > UCL for a surrogate, apply J to all positive results  if the %R < LCL for a surrogate, apply J to all positive results; apply R to all non-detect results  If any surrogate recovery is <10%, apply R to all results
		MDL study	Once per 12 month period	Detection limits established shall be ≤ ½ the RLs in Table 6.2.5-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	None	none	none	Apply F to all results between MDL and RL

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
	Semi-volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF $\geq$ 0.30°; and %RSD for CCCs < 30%; and %RSD for all other calibration analytes $\leq$ 15%*	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Second- source calibration verification	Once per five- point initial calibration	All analytes within ±25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
SW8270 C		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF $\geq$ 0.05; and CCCs $<$ 20% drift; and all calibration analytes within $\pm$ 20% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 6.2.6-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description (section 6.0)	Retune instrument and verify	Apply R to all results for all samples associated with the tune

	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Actiona	Criteria <sup>b</sup>
	Semi-volatile Organics	Iss	Immediately after or during data acquisition of calibration check standard	Retention time ±30 seconds: EICP area within -50% to +100% of last calibration verification (12 hours) for each	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Apply R to all results for specific analytes for all samples associated with the IS
		Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
SW8270 C		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 6.2.6-2	Correct problem then reprep and analyze the LCS and all samples in the affected USACE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects
	Semi-volatile Organics	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 6.2.6-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply J to all positive results, apply R to all non-detects
						If any surrogate recovery is < 10%, apply R to all results

	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Action <sup>a</sup>	Criteria <sup>b</sup>
SW8270 C	Z GLAMOOT	MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 6.2.6-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if;(1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3) MS/MSD RPD > CL
		MDL study	Once per 12 month period	Detection limits established shall be < the RLs in Table 6.2.6-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	None	none	none	Apply F to all results between MDL and RL
	PAHs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD < 20% for CFs or RFs	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
SW8310		Second- source calibration verification	Once per five- point initial calibration	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Retention time window calculated for each analyte	Each initial calibration and calibration verifications	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
		Initial calibration verification	Daily, before sample analysis	All analytes within ±15% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Continuing calibration verification	After every 10 samples and at the end of the analysis sequence	All analytes within ±15% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification	Apply R to all results for the specific analyte(s) in all samples since the last acceptable calibration verification
SW8310		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria, Table 6.2.7-2	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
	PAHs	Method blank	One per analytical batch	No analytes detected ≥ RL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, Table 6.2.7-2	Correct problem then reprep and analyze the LCS and all samples in the affected USACE analytical batch	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results if the LCS %R < LCL, apply J to all positive results, apply R to all non-detects

36.41.1	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	<b>Action</b> <sup>a</sup>	Criteria <sup>b</sup>
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria, Table 6.2.7-2	Correct problem then reextract and analyze sample	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply J to all positive results, apply R to all non-detects If any surrogate recovery is < 10%, apply R to all results
SW8310	PAHs	MS/MSD	One MS/MSD per every 20 project samples per matrix	QC acceptance criteria, Table 6.2.7-2	none	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if; (1)%R for MS or MSD > UCL or (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL
		Confirmatio n <sup>c</sup>	100% for all positive results	Same as for initial or primary analysis	Same as for initial or primary analysis	Apply R to the result for the specific analyte(s) in the sample
		MDL study	Once per 12 month period	Detection limits established shall be < the RLs in Table 6.2.7-1	none	Apply R to all results for the specific analyte(s) in all samples analyzed
		Results reported between MDL and RL	none	none	none	Apply F to all results between MDL and RL

Method	Applicable	QC Check	Minimum	Acceptance	Corrective	Flagging
	Parameter	,	Frequency Initial	Criteria	Action <sup>a</sup>	Criteria <sup>b</sup>
	Total Petroleum Hydrocarbon	Five-point initial calibration for all analytes	calibration prior to sample analysis	Mean RSD for TPH 25% or correlation coefficient for linear regression of 0.995.	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
TX1005		Calibration verification	At the beginning of each working day in which TPH analysis will occur and at the end of each batch of 20 samples, each shift, or each work day, whichever is more frequent.	RPD – 25% of expected value	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Initially prior to analysis of any samples and in response to changes in staff, instrumentation or operations	Average %R within 75 – 125% and RSD – 20% or within laboratory limits	Recalculate results; locate and correct problem with system and then rerun demonstration for those analytes that did not meet criteria	Apply R to all results for all samples analyzed by the analyst
		Method blank	One per analytical batch of 20 samples or less	No analytes detected - MQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
TX1005		LCS and LCSD	One LCS/LCSD per analytical batch of 20 samples or less	%R within 75 – 125% and RPD ± 20% or within the laboratory limits	Correct problem then reprep and analyze the LCS/LCSD and all samples in affected analytical batch	For analyte(s) in all samples in the associated analytical batch; if the LCS/LCSD %R > UCL, apply J to all positive results. If the LCS/LCSD %R < LCL, apply J to all positive results, apply J to all positive results, apply R to all non-detects
		MS and MSD	One MS/MSD per every 20 project samples per matrix	%R within 75 – 125% and RPD ± 20% or within laboratory limits	None	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if;  (1) %R for MS or MSD > UCL or  (2) %R for MS or MSD < LCL or  (3) MS/MSD RPD> CL

	Applicable GGG , Minimum Acceptance Corrective Flagging					
Method	Applicable Parameter	QC Check	Frequency	Acceptance Criteria	Corrective Action <sup>a</sup>	Flagging Criteria <sup>b</sup>
TX1005	Total Petroleum Hydrocarbon s	Surrogate Recoveries	Spiked into every sample including all QC	% R within 70 – 130% or laboratory established limits	Reanalyze, or reextract and reanalyze, or flag the data	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply R to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results
		Retention Time window check	Once per analytical batch of 20 samples or less	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		MDL study	Once per 12 month period	Within manufacturer's statistical guidelines	none	none
		Performance Evaluation samples	Once per 12 month period	Detection limits established shall be ½ the MQLs	None	Reextract/reanaly zed. If still out, correct problem and reanalyze
TX1006	Characterizat ion of Nc6 to Nc35 Petroleum Hydrocarbon s	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Mean RSD for TPH 25% or correlation coefficient for linear regression of 0.995	Correct problem then repeat initial calibration	Apply R to all results for specific analyte(s) for all samples associated with the calibration

	Applicable	0.0.0	Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	Action <sup>a</sup>	Criteria <sup>b</sup>
		Calibration verification  Demonstrate	At the beginning of each working day in which TPH analysis will occur and at the end of each batch of 20 samples, each shift, or each work day, whichever is more frequent.  Initially prior to	RPD – 25% of expected value  Average %R	Correct problem then repeat initial calibration  Recalculate results;	Apply R to all results for specific analyte(s) for all samples associated with the calibration  Apply R to all
TX1006		ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	analysis of any samples and in response to changes in staff, instrumentation or operations	within 75 – 125% and RSD – 20% or within laboratory limits	locate and correct problem with system and then rerun demonstration for those analytes that did not meet criteria	results for all samples analyzed by the analyst
		Method blank	One per analytical batch of 20 samples or less	No analytes detected < MQL	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank	Apply B to all results for the specific analyte(s) in all samples in the associated analytical batch
		Aliphatic and Aromatic Fractionatio n Check Standards	One per each batch of silica gel	%R within 60 – 140% and < 10 – 20 % crossovers		

35.0	Applicable		Minimum	Acceptance	Corrective	Flagging
Method	Parameter	QC Check	Frequency	Criteria	<b>Action</b> <sup>a</sup>	Criteria <sup>b</sup>
TX1006		MS and MSD	One LCS per analytical batch of 20 samples or less  One MS/MSD per every 20 project samples per matrix	%R within 60 – 140% and RPD ± 20% or within the laboratory limits  %R within 60 – 140% and RPD ± 20% or within laboratory limits	Correct problem then reprep and analyze the LCS and all samples in affected analytical batch  None	For analyte(s) in all samples in the associated analytical batch; if the LCS %R > UCL, apply J to all positive results. If the LCS %R < LCL, apply J to all positive results, apply R to all non-detects  For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M if;  (1) %R for MS or MSD > UCL or (2) %R for MS or MSD < LCL or (3) MS/MSD RPD > CL

Method	Applicable	QC Check	Minimum	Acceptance	Corrective	Flagging
Michiga	Parameter		Frequency	Criteria	Actiona	Criteria <sup>b</sup>
TX1006	Characterizat ion of Nc6 to Nc35 Petroleum Hydrocarbon s	Surrogate Recoveries	Spiked into every sample including all QC	% R within 70 – 130% or laboratory established limits	Reanalyze, or reextract and reanalyze, or flag the data	For the samples; if the %R > UCL for any surrogate, apply J to all positive results if the %R < LCL for any surrogate, apply J to all positive results, apply J to all positive results, apply R to all non-detects  If any surrogate recovery is < 10%, apply R to all results
		Retention Time window check	Once per analytical batch of 20 samples or less	± 3 times standard deviation for each analyte retention time from 72-hour study	Correct problem then reanalyze all samples analyzed since the last retention time check	Apply R to all results for the specific analyte(s) in the sample
		MDL study	Once per 12 month period	Within manufacturer's statistical guidelines	none	none
		Performance Evaluation samples	Once per 12 month period	Detection limits established shall be ½ the MQLs	None	Reextract/reanaly zed. If still out, correct problem and reanalyze

#### **Data Qualifiers**

Qualifier	Description
J	The analyte was positively identified, the quantitation is an estimation.
U	The analyte was analyzed for, but not detected. The associated numerical value is at or below the MDL.
F	The analyte was positively identified but the associated numerical value is below the RL.
R	The data are unusable due to deficiencies in the ability to analyze the sample and meet QC criteria.
В	The analyte was found in an associated blank, as well as in the sample.
М	A matrix effect was present.
S	To be applied to all field screening data.
Т	Tentatively identified compound (using GC/MS)